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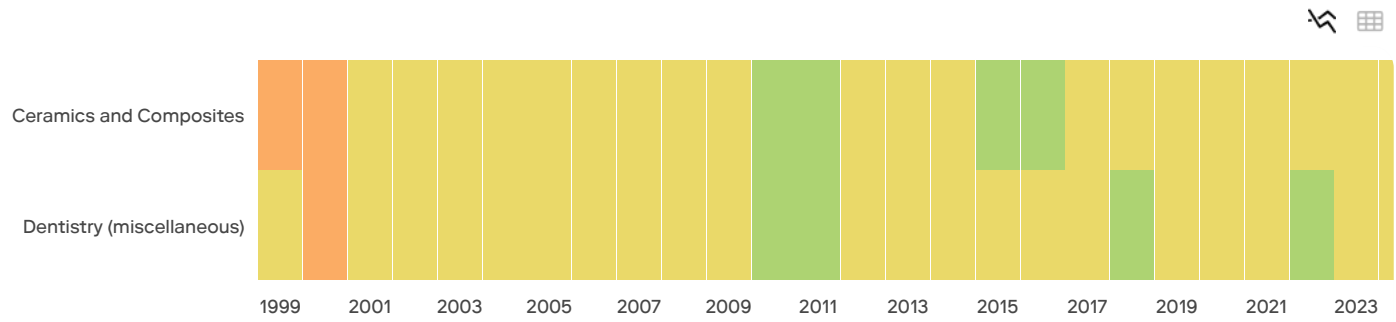

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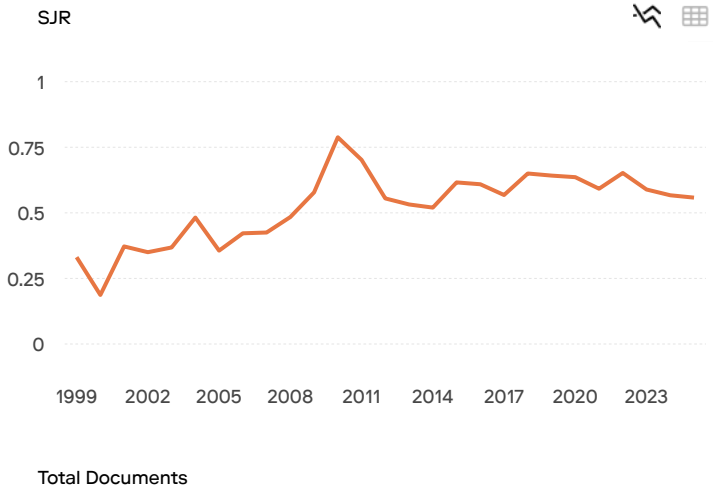

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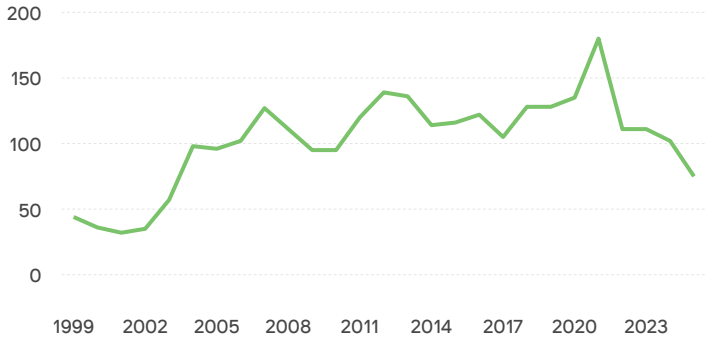

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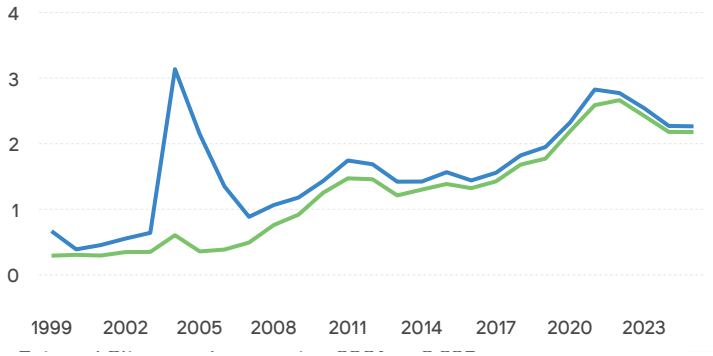

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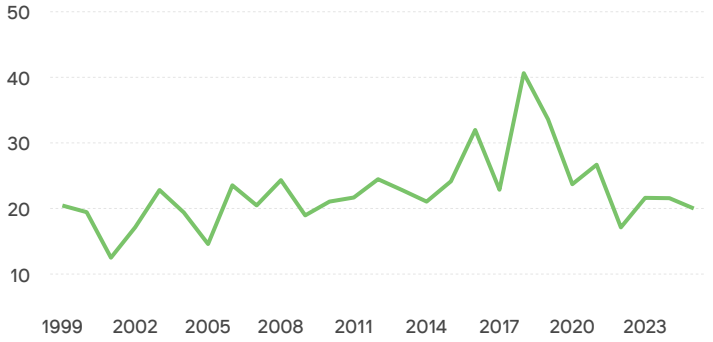

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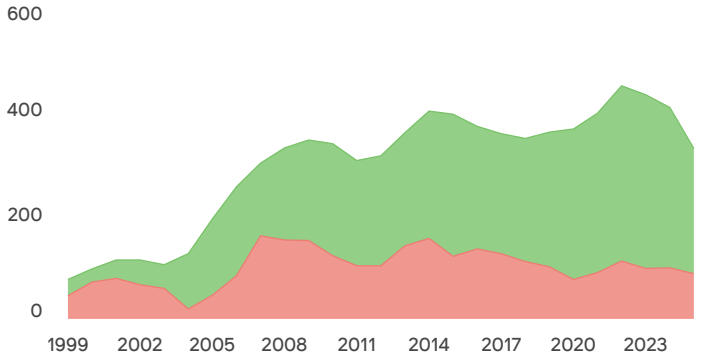

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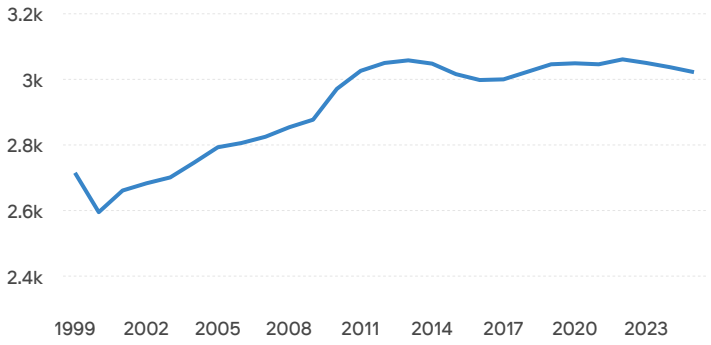

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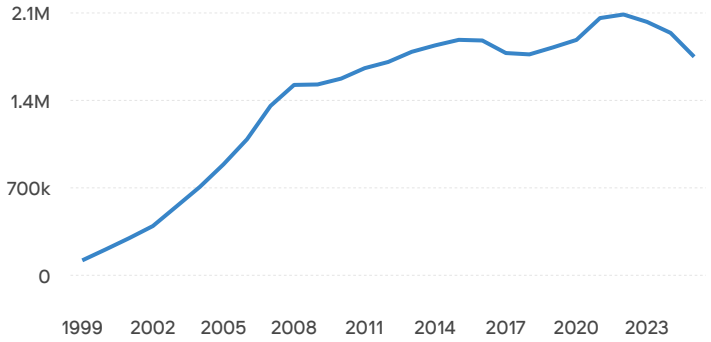

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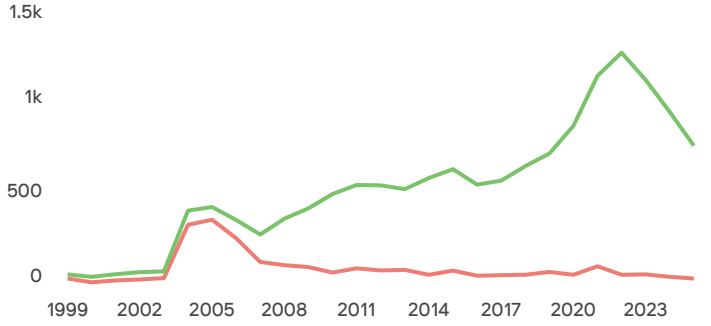

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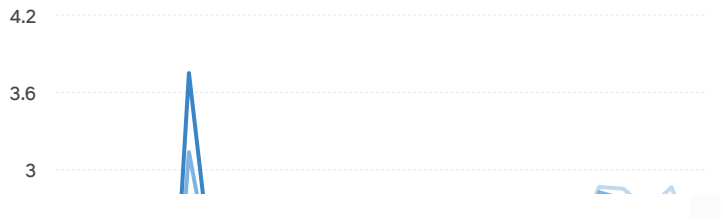

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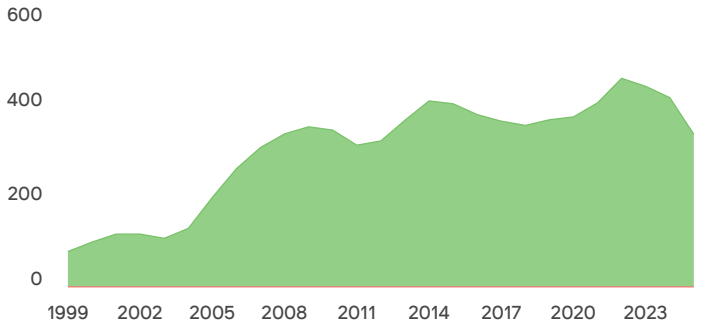

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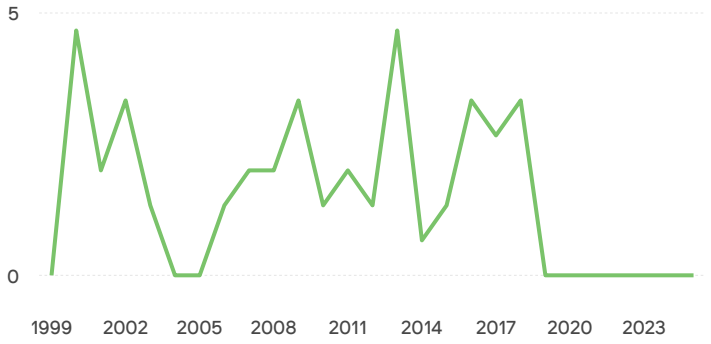

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Flexural Performance of Flowable *versus* Conventional Light-cured Composite Resins in a Long-term *in vitro* Study

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The purpose of this study was to evaluate the flexural strength, flexural modulus, modulus of resilience, and water sorption of four flowable light-cured composite resins (FCRs). Results were then compared with four conventional composite resins (CCRs) and a minifilled hybrid light-cured composite resin, which served as a control. Twenty specimens were flexural tested immediately after curing, while others were stored in water at 37℃ for 1 month, 3 months, 6 months, or 1 year before flexural testing. The 1-year specimens were weight-measured at designated time intervals to examine water sorption. All FCRs, except Point 4 Flowable, showed higher flexural strength values than their CCR counterparts ($p<0.05$, Scheffé's test). After one-year water storage, the flexural strengths and flexural moduli of FCRs increased 1.5-fold or more when compared with the immediate condition. In most cases, the moduli of resilience of FCRs were higher than those of CCRs. In conclusion, it was found that FCR and CCR with the same brand name had very different characteristics and mechanical properties.

Keywords: Flowable composite, Mechanical property, Long-term durability

INTRODUCTION

Resin composite formulations with easy-to-flow characteristic have been introduced as the so-called 'flowable' composite resins (FCRs). Bayne *et al*. reported that the mechanical properties of several commercial FCRs were about 60% to 90% of those of conventional composite resins (CCRs)[1]. Therefore, FCR is usually used as a liner in areas of difficult access or flow such as irregular internal surfaces and proximal boxes of Class II preparations. This is because FCR easily flows into, adapts to, and fills the tooth surface, resulting in less leakage and hence less internal restorations with voids and post-operative sensitivity[1-7]. However, in restorations prepared by sandwich technique, gaps still exist between the FCR liner and the hybrid composite filling material[8].

Despite limited scientific information, FCRs are used in a wide range of applications[9,10]. Nonetheless, dentists are uncertain and divided if FCR can be used as a complete restoration. This is because caution is already exercised on the amount of FCR to be used in posterior restorations to avoid problems arising from the poor mechanical properties of FCRs[8]. Meanwhile, owing to strong tides of continuous research development and high demand for FCRs, some manufacturers have ridden on the success of their CCR products and launched the FCR products. Despite the deluge of FCR products that have entered the market, no studies have been undertaken to compare FCRs with their CCR counterparts of the same brand name.

In the oral environment, FCRs must withstand masticatory and parafunctional stresses. They must maintain their integrity while transferring stresses to the tooth structure. It is also noteworthy that these stresses vary markedly in different clinical situations. Consequently, threshold values for mechanical properties — which were suggested to ensure long-term success of dental restorations — may vary considerably from one case to another. In cases where greater stresses are anticipated, then stronger FCRs are required. Therefore, with a view to unraveling the real intrinsic characteristics of FCRs, it is preferable to measure the strength of the FCR *per se*, without being placed in the tooth cavity and thus inviting the complicating presence of adhesive materials.

To determine if a composite resin filling material can resist masticatory force, flexural strength is one of the mechanical properties that must be assessed[11-13]. As a collective measurement of tensile, compressive and shear stresses simultaneously, flexural strength measurement is used to evaluate the fracture resistance and elasticity of a material[12,13]. Then, for the analysis of flexural strength data, Weibull distribution analysis has been recommended. This is because it is capable of allowing for skewed data and predicting values within and outside the data set[4,14-19]. By measuring flexural strength, the

flexural modulus and modulus of resilience are measured too. Flexural modulus describes the stiffness of a material, since marginal breakdown and loss of marginal seal are most likely to occur in products with a lower modulus of elasticity[1,17,20]. Then, the energy needed to break a material is expressed by the modulus of resilience[11,22,23].

The flexural properties of composite resins are also time-dependent. As the polymerization of composites progresses even after being light-cured[17,23], an incomplete polymerization may predispose resin restoratives to degradation. Unreacted molecules can form walls of pores within the bulk material, which can be filled with water and cause water sorption[11]. However, this characteristic can be advantageous to FCR as a partial compensation for polymerization shrinkage[24]. For this reason, the long-term flexural characteristics of FCR must be studied because of important clinical implications. Besides, physical properties — such as water sorption that plays an important role in the long-term success of restorative materials — must be considered too[22,25].

Therefore, this investigation was carried out to evaluate the long-term flexural strength, flexural modulus, modulus of resilience, and water sorption of FCR. The hypothesis tested was that the properties of FCR would be significantly different from those of CCR.

MATERIALS AND METHODS

Materials used

Four FCRs (Metafil Flo, Filtek Flow, Unifil Flow, and Point 4 Flowable) and four CCRs (Metafil C, Filtek

Table 1 Materials investigated in this study. Information is as provided by the manufacturers

Material (Manufacturer)	Batch no.	Filler content	Monomer	Curing time (seconds)
Metafil Flo* (Sun Medical, Moriyama, Japan)	VV10, VV12, EK1, EK2	Barium silica glass, colloidal silica & TMPT Filler content: 44 vol% (65 wt%) Filler particle size: 0.01−10 μm	UDMA	40
Metafil C** (Sun Medical, Moriyama, Japan)	TE1	TMPT & colloidal silica Filler content: 54 vol% (66 wt%) Filler particle size: 0.01−10 μm	UDMA	40
Filtek™ Flow* (3M, St. Paul, MN, USA)	OBK, 20010104	Silica & silica zirconia Filler content: 47 vol% (68 wt%) Filler particle size: 1.50 μm	Bis-GMA, TEGDMA	20
Filtek A 110** (3M, St. Paul, MN, USA)	1AP	Inorganic silica Filler content: 40 vol% (56 wt%) Filler particle size: 0.04 μm	Bis-GMA	40
Point 4 Flowable* (Kerr, Orange, CA, USA)	206B43	Barium silica glass Filler content: 48 vol% (70 wt%)	TEGDMA, EBPADM	40
Point 4** (Kerr, Orange, CA, USA)	205553	Barium aluminoborosilicate glass Filler content: 57 vol% (76 wt%) Filler particle size: 0.4 μm	Bis-GMA, TEGDMA & EBPADM	40
Unifil Flow* (GC Corp. Tokyo, Japan)	0107201	Fluoro-alumino-silicate, silica Filler content: 67 wt% (vol%: Not Available) Filler particle size: 0.7 μm	UDMA (26%) Dimethacrylate (7%)	40
Unifil F** (GC Corp. Tokyo, Japan)	161181, 0204031	Fluoro-alumino-silicate, silica Filler content: 77 wt% (vol%: Not Available) Filler particle size: 0.8−0.9 μm	UDMA (16%) Dimethacrylate (7%)	20
Herculite XRV** Kerr, Orange, CA, USA	112330	Barium silicate Filler content: 59 vol% (78.8 wt%) Filler particle size: 0.6 μm	Bis-GMA, TEGDMA	40

* : Flowable type (FCR), ** : Conventional type (CCR) ; UDMA=Urethane dimethacrylate; Bis-GMA=Bisphenyl glycidyl-methacrylate ; TEGDMA=Triethylene glycol-dimethacrylate; EBPADM=Ethoxylated bis-phenol A dimethacrylate

A110, Unifil F, and Point 4) of Shade A3, paired from the same manufacturers, were used in this study. A minifilled hybrid light-cured composite resin (Herculite XRV) was used in this study as a control material. Details of the materials, as provided by the manufacturers, are listed in Table 1.

A visible light curing unit (New Light VL-II, GC Corp., Tokyo, Japan) with an irradiated diameter of 10 mm was used for activating the specimens. Close contact between exit window of the lamp and celluloid strip was ensured. Light intensity was checked and maintained at 450 mW/cm^2 using a radiometer (Demetron/Kerr, Danbury, CT, USA).

Flexural strength measurement

Internal dimensions of the Teflon split molds used in this study were 25×2×2 mm. A total of 100 specimens were prepared for each material. Each mold was filled with the material, covered with a celluloid strip, and then the glass plate clamped. After 15 seconds, the glass plate was removed and the specimen was light-cured in three overlapping sections with an irradiation time of 20, 30, or 40 seconds according to manufacturer's recommendations. After the specimens were removed from the molds, excess material was removed with a silicon carbide bur. Following which, the specimens were polished with #600 sandpaper to acquire flat surfaces.

Twenty specimens were flexural tested immediately, while others were immersed in 37℃ distilled water in an incubator for the following storage periods before flexural testing: 1 month, 3 months, 6 months, or 1 year. For each material and the designated storage period, 20 specimens were prepared. Prior to testing, the specimens were measured using a digital micrometer (No. 293-421-20, Mitsutoyo, Kawasaki, Japan).

Using a three-point bending method with a 20-mm span and a crosshead speed of 0.5 mm/min, flexural strength was measured by mounting the apparatus on a universal testing machine (Autograph DCS-2000, Shimadzu, Kyoto, Japan) as outlined in ISO 9917-2 (1996)[22,26]. Then, a maximum external force of 10 kgf (98 N) was applied to the middle of the test beam.

From the flexural strength result, flexural modulus in MPa was calculated and subsequently converted to GPa[9,18,23]. Then, using the values of flexural strength and flexural modulus, the modulus of resilience was computed[11,21].

All procedures, except for mechanical testing, were performed in an air-conditioned room at 23±0.5℃ and 50±2% relative humidity.

Change in weight after 24 hour/water sorption

To assess the degree of water sorption, specimens for 1-year flexural strength measurement were weighed — prior to immersion in distilled water — with an electric balance (AJ 100, Mettler, Greifensee, Switzerland). The specimens were weighed again after being stored in 37℃ distilled water for 24 hours and at several time intervals up to one year. Prior to weight measurement, the specimens were dried for one minute on Kim Wiper. Changes in specimen's weight between the immediate condition (*i.e.*, immediately after light curing) and after water storage were expressed as a percentage[22].

Statistical analysis

Flexural strength and flexural modulus results were analyzed statistically using three-way ANOVA and Scheffé's test[27] at a significance level of 0.05. Flexural strength test results were also analyzed statistically using Weibull statistics with the following equation:

$$Pf = 1 - \exp[-(\sigma / \sigma_o)m]$$

where Pf is the probability of failure, σ is the strength at a given Pf, σ_o is the characteristic strength, and m is the Weibull modulus, a constant factor related to the dispersion of failure data[14,15,17].

Results of water sorption test were also analyzed statistically using three-way ANOVA and Scheffé's test[27]. Subsequently, using Sigma Plot 8.0 program (SPSS, Chicago, IL, USA), the data were fitted to a non-linear curve by plotting water sorption (percentage) as a function of time (days).

RESULTS

Flexural strength

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the flexural strength of composite resins significantly ($p<0.001$). Table 2 lists the flexural strength results of all the materials tested in this study. Scheffé's test between the pairs and among the materials within the same water storage period showed significant differences ($p<0.05$). In the immediate condition, all FCRs would be completely fractured by a flexural stress of 90 MPa, with the mean value of Point 4 Flowable > Metafil Flo > Unifil Flow > Filtek Flow. All FCRs, except Point 4 Flowable, showed higher flexural strength values than their CCR counterparts, and that this situation continued up to one year. Predicting from Weibull cumulative Gaussian plots, such as those of Unifil pair and Herculite XRV (Fig. 1), it could be seen that for all 1-year FCR specimens, a minimum of 110 MPa flexural stress must be applied before fracture would occur. On the contrary, 110 MPa was the maximum strength required for fracturing the CCRs.

Weibull analysis (Table 3) showed that Weibull moduli varied from 5.01 to 14.30. Except for Point 4 Flowable, the characteristic strengths of CCRs were lower than those of FCRs. Further, statistical analysis showed high coefficients of correlation (r), varying between 0.96 and 0.98.

Flexural modulus

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the flexural modulus of composite resins significantly ($p<0.001$). In the immediate condition, as shown in Table 4, the flexural moduli of FCRs were in the range of 1.38−3.16 GPa. Those of CCRs were between 2.03 and 3.71 GPa, and that of control was 5.62 GPa. After one year, the flexural moduli increased. The flexural moduli of FCRs increased to a range of 5.51−7.47 GPa, while those of their CCR counterparts were between 4.53 and 8.89 GPa, and that of Herculite XRV was 9.50 GPa. Scheffé's test showed significant differences in flexural modulus within each CCR-FCR pair ($p<0.05$). In general, the flexural moduli of Unifil F, Filtek A110, and Herculite XRV were higher than those of Unifil Flow and Filtek Flow. However, in most conditions, the flexural modulus of Metafil C was lower than that of Metafil Flo.

Modulus of resilience

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the modulus of resilience of composite resins significantly ($p<0.001$). As shown in Table 5, Scheffé's test for the modulus of resilience showed that FCRs had higher modulus of resilience values than CCRs. Further, throughout the one-year period, all FCRs showed almost no significant differences among their values. The moduli of resilience of FCRs varied between 0.62 MJ/m^3 and 1.93 MJ/m^3, while those of CCRs varied between 0.20 MJ/m^3 and 1.21 MJ/m^3.

Change in weight after 24 hours/water sorption

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the water sorption of composite resins significantly ($p<0.001$). The results of water sorption are listed in Table 6. After one-year storage, all FCRs had water sorption values between 0.39% and 1.18%, while the CCRs showed water sorption values between 0.98% and 2.96%. All FCRs, except Point 4 Flowable, showed significantly lower water sorption values than their CCRs ($p<0.05$), and that Unifil F showed the highest water sorption value among the CCRs (Fig. 2). The water

Table 2 Mean values of flexural strength of composite resins (Mean(SD) in MPa)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	75.69 (7.35) $^{C,\omega,\zeta}$	127.27 (14.35) $^{B,\rho,\sigma}$	119.91 (12.77) $^{B,\kappa,\lambda}$	77.73 (8.65) $^{C,\iota}$	142.37 (12.47) $^{A,\alpha}$
Metafil C**	50.10 (60.2) $^{E,\#}$	76.91 (4.22) $^{C,\tau}$	67.56 (8.19) $^{C,D,o,\pi}$	60.81 (8.11) $^{D,E,\phi}$	75.82 (10.46) $^{C,\zeta,\delta}$
Filtek ™ Flow*	62.42 (10.29) $^{G,\Psi,\zeta}$	111.25 (13.44) $^{F,\sigma}$	106.08 (11.94) $^{F,\mu}$	113.33 (13.23) $^{F,\gamma,\eta}$	112.59 (9.90) $^{F,\beta}$
Filtek A 110**	55.44 (7.70) $^{G,\zeta,\#}$	52.52 (5.82) $^{G,\nu}$	51.09 (7.63) $^{G,\pi,\theta}$	61.01 (6.17) $^{G,\phi}$	48.71 (5.91) ^{G,s}
Point 4 Flowable*	86.88 (11.46) $^{I,\varpi,\omega}$	124.63 (17.90) $^{H,\rho,\sigma}$	88.69 (14.02) $^{I,\mu,\nu}$	90.63 (12.19) $^{I,\eta,\iota}$	88.83 (11.77) $^{I,\delta}$
Point 4**	88.02 (13.22) $^{I,\varpi}$	129.12 (16.68) $^{H,\rho}$	129.22 (20.29) $^{H,\kappa}$	117.59 (16.40) $^{H,\gamma}$	118.52 (20.16) $^{H,\beta}$
Unifil Flow*	71.37 (5.93) $^{L,M,\xi,\Psi}$	126.70 (13.59) $^{K,\rho,\sigma}$	132.30 (20.46) $^{K,\kappa}$	136.82 (16.97) $^{J,K,\phi}$	153.08 (11.90) $^{J,\alpha}$
Unifil F**	67.88 (10.37) $^{M,\xi,\Psi}$	87.55 (17.31) $^{L,\tau}$	78.37 (12.80) $^{L,M,\nu,o}$	60.86 (12.82) $^{M,\phi}$	68.02 (11.72) $^{M,\delta}$
Herculite XRV**	90.20 (11.28) $^{R,\varpi}$	119.20 (15.46) $^{P,\rho,\sigma}$	108.38 (15.96) $^{P,Q,\lambda}$	97.22 (16.77) $^{Q,R,\eta}$	115.29 (18.99) $^{P,\beta}$

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin

Identidal capital letters indicate no significant difference ($p>0.05$, Scheffé's test) analyzed between pairs and among the values of control

Identical Greek letters and # indicate no significant difference ($p>0.05$, Scheffé's test) analyzed among the values of the same period

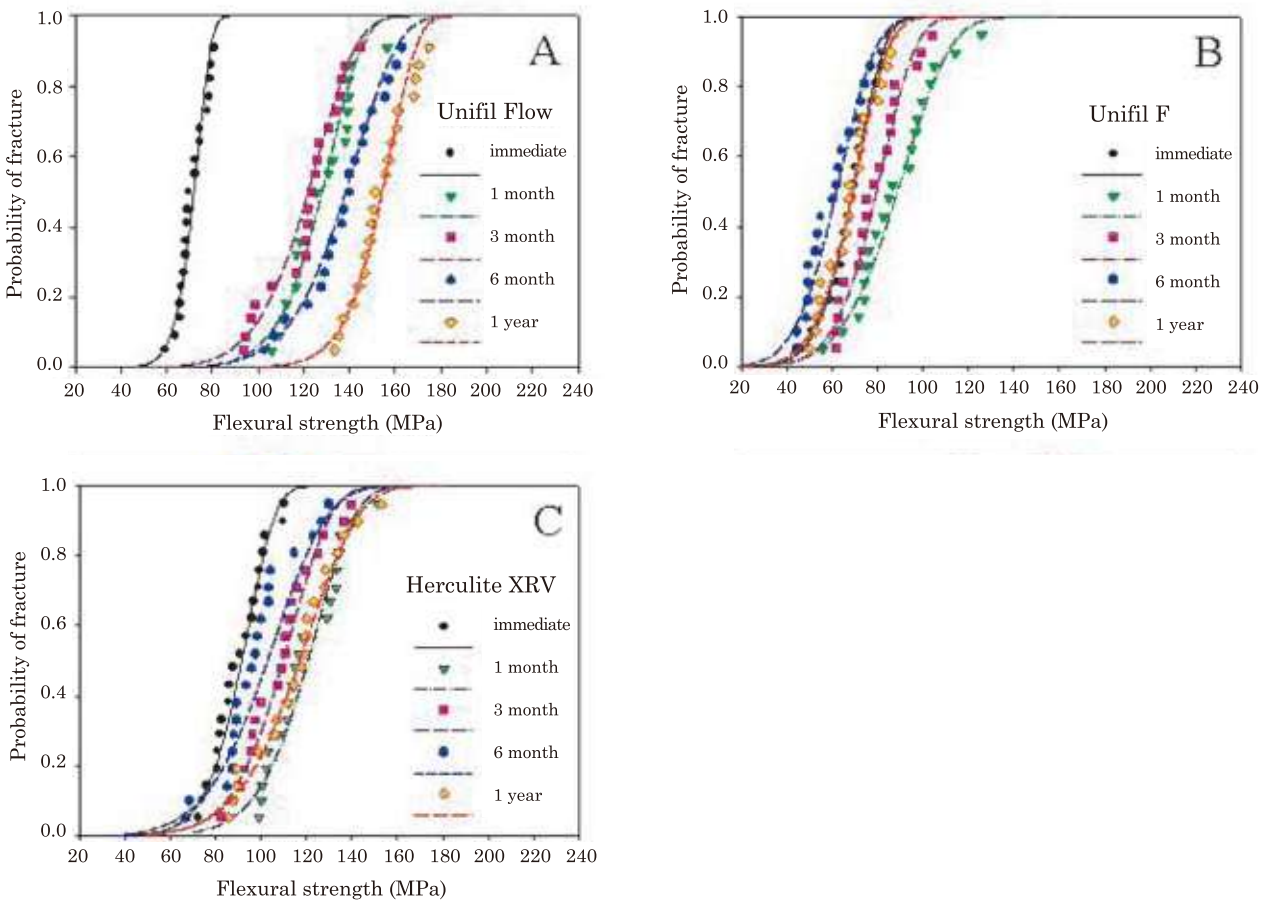

Fig. 1 Weibull cumulative Gaussian plots for Unifil pair and Herculite XRV.

Table 3 Characteristic strengths (MPa) and Weibull moduli of composite resins

Material	Immediate		1 month		3 month		6 month		1 year	
	CS	WM	CS	WM	CS	WM	CS	WM	CS	WM
Metafil Flo*	79.08	10.35	133.69	9.47	125.67	9.98	82.51	8.68	148.16	12.04
Metafil C**	52.80	8.82	78.59	10.98	71.30	8.62	64.69	7.34	80.51	7.57
Filtek ™ Flow*	66.89	6.37	117.73	8.18	111.44	9.45	119.26	9.10	117.08	12.09
Filtek A 110**	58.90	7.47	55.14	9.58	54.40	7.16	63.88	10.25	51.45	8.41
Point 4 Flowable*	92.02	7.93	132.41	7.45	94.82	6.64	96.21	7.62	94.08	7.97
Point 4**	93.87	6.96	136.47	8.29	138.04	6.71	124.81	7.58	127.21	6.20
Unifil Flow*	74.13	12.70	132.94	9.80	141.19	6.85	144.52	8.38	158.58	13.73
Unifil F**	72.49	6.75	94.74	5.36	83.95	6.44	66.18	5.01	73.10	6.06
Herculite XRV**	95.22	8.50	126.21	8.06	115.30	7.26	109.69	5.54	123.71	6.34

n=20 ; CS=characteristic strength; WM=Weibull modulus
*=flowable light-cured composite resin; **=conventional light-cured composite resin

Table 4 Mean values of flexural modulus of composite resins (Mean(SD) in GPa)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	1.95 (0.22) E, δ, ε	5.04 (0.42) B, κ	5.06 (0.43) B, θ, ρ	3.31 (0.23) D, ξ	7.47 (0.67) A, ζ
Metafil C**	2.03 (0.17) E, δ	4.36 (0.31) C, κ, λ	4.02 (0.28) C, σ	3.42 (0.20) D, ξ	4.53 (0.27) B, C, ¥
Filtek ™ Flow*	1.38 (0.09) M, φ	4.32 (0.26) H, λ	4.55 (0.41) I, ρ, σ	4.87 (0.74) H, ω	5.51 (0.43) I, J, $
Filtek A 110**	3.55 (0.28) L, β, δ	7.12 (0.30) K, ι	6.17 (0.57) K, π	6.99 (0.26) K, ϖ	6.05 (0.11) I, #, $
Point 4 Flowable*	3.16 (0.38) N, δ	6.39 (0.51) T, φ	5.65 (0.35) U, π, θ	6.68 (0.41) S, T, ϖ	6.45 (0.48) S, T, #
Point 4**	3.71 (0.40) N, β	7.68 (0.65) Q, R, ι	7.10 (0.61) R, S, o	8.15 (0.67) Q, ν	8.87 (0.59) P, Ψ
Unifil Flow*	1.47 (0.27) Z, S, φ	4.98 (0.57) Y, κ	5.34 (0.85) X, Y, θ	5.52 (0.45) X, Y, ω	6.24 (0.62) X, #
Unifil F**	2.24 (0.27) Z, δ	8.49 (0.92) W, η	8.34 (0.99) W, ν	9.84 (1.19) V, τ	8.89 (0.82) W, Ψ
Herculite XRV**	5.62 (0.50) F, α	9.60 (0.61) G, γ	9.54 (0.54) G, μ	9.18 (0.93) G, τ	9.50 (0.51) G, Ψ

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant difference (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters and symbols ($, ¥, #) indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values of the same period

Table 5 Mean values of the modulus of resilience of composite resins (Mean(SD) in MJ/m^3)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	1.50 (0.31) A, α, β	1.65 (0.38) A, φ	1.45 (0.37) A, κ, λ	0.93 (0.18) B, ρ	1.35 (0.28) A, ξ
Metafil C**	0.65 (0.12) B, C, ε	0.68 (0.11) B, C, ι	0.58 (0.15) C, μ, ν	0.55 (0.14) C, τ	0.69 (0.14) B, C, Ψ
Filtek ™ Flow*	1.50 (0.50) E, F, α, β	1.52 (0.19) E, φ, γ	1.41 (0.28) E, F, λ	1.35 (0.06) E, F, θ	1.20 (0.26) F, ξ
Filtek A 110**	0.45 (0.10) G, ε	0.20 (0.03) G, φ	0.24 (0.06) G, o	0.28 (0.04) G, ν, π	0.20 (0.03) G, ζ
Point 4 Flowable*	1.24 (0.43) H, I, β, π	1.25 (0.38) H, γ, η	0.80 (0.17) J, K, μ	0.63 (0.17) K, σ, τ	0.62 (0.18) K, Ψ
Point 4**	1.05 (0.33) H, I, J, K, π, δ	1.13 (0.37) H, I, J, η	1.21 (0.37) H, I, j, λ	0.86 (0.22) I, J, K, ρ, σ	0.81 (0.29) J, K, Ψ
Unifil Flow*	1.82 (0.37) L, a	1.66 (0.47) L, φ	1.74 (0.25) L, κ	1.71 (0.36) L, π	1.93 (0.39) L, ω
Unifil F**	1.07 (0.36) M, β, π, δ	0.47 (0.18) N, ι, φ	0.38 (0.15) N, ν, o	0.22 (0.10) N, ϖ	0.27 (0.08) N, ζ
Herculite XRV**	0.74 (0.20) P, Q, δ, ε	0.75 (0.20) P, ι	0.63 (0.20) P, Q, μ, ν	0.54 (0.21) Q, τ, ν	0.72 (0.24) P, Q, Ψ

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant dirrence (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values same period

Table 6 Mean values of water sorption of composite resins (Mean(SD) in %)

Material	1 month	3 month	6 month	1 year
Metafil Flo*	0.37 (0.04) A, γ	0.38 (0.03) A, ν	0.39 (0.03) A, ϖ	0.39 (0.03) A, #
Metafil C**	1.48 (0.10) B, δ	1.48 (0.10) B, φ	1.50 (0.09) B, θ	1.50 (0.09) B, Ψ
Filtek ™ Flow*	0.96 (0.40) G, ε	0.97 (0.05) G, λ	0.99 (0.05) G, σ	0.99 (0.04) G, ¥
Filtek A 110**	1.58 (0.11) L, β	1.69 (0.12) L, ι	1.84 (0.10) K, π	1.98 (0.14) K, ξ
Point 4 Flowable*	0.72 (0.04) Q, R, φ	0.80 (0.08) P, Q, μ	0.86 (0.07) N, P, τ	0.93 (0.08) M, N, ¥
Point 4**	0.66 (0.55) R, φ	0.77 (0.04) Q, μ	0.79 (0.06) P, Q, τ, υ	0.98 (0.05) M, ¥
Unifil Flow*	1.14 (0.04) V, δ	1.17 (0.04) V, κ	1.18 (0.03) V, ρ	1.18 (0.03) V, ζ
Unifil F**	2.81 (0.09) Y, α	2.95 (0.13) Z, η	2.95 (0.13) Z, o	2.96 (0.11) Z, ω
Herculite XRV**	0.64 (0.05) C, φ	0.70 (0.06) C, D, μ	0.74 (0.08) D, F, υ	0.80 (0.07) F, $

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant difference (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters and symbols ($, ¥, #) indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values of the same period

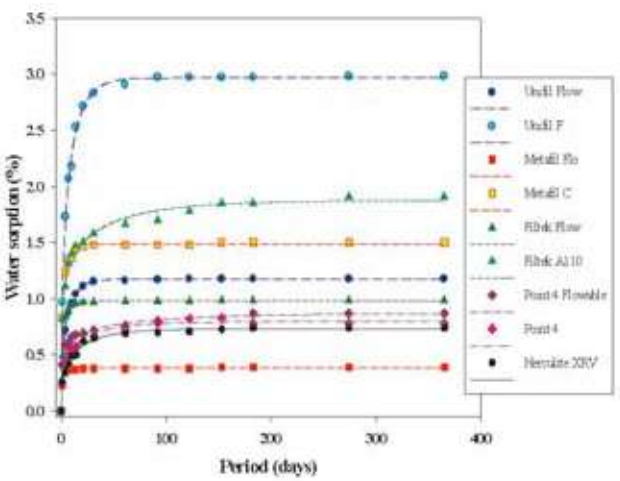

Fig. 2 Water sorption trends of FCRs and CCRs during one year of water storage.

sorption values correlated with the period of water immersion time and could be fitted to a sigmoidal curve of Chapman's three-parameter equation [$y = a\times(1-exp(-bx))^c$] or Hill's three-parameter equation [$y = ax^b/(c^b + x^b)$], with coefficient of correlation r≥0.997.

DISCUSSION

Measurement of long-term flexural strength is very important for a composite resin, since it describes the durability of the material. The degradation of composite resins in the oral environment is considered to be attributed to the resin matrix, filler particles, and hydrolytic instability of silane coupling agent at the polymer-silica interface[28-35]. After one-year water storage in the present study, all composites showed higher flexural strengths in comparison with their immediate values. FCRs increased their strengths by up to 2.14-fold, whereas their CCR counterparts increased by only up to 1.54-fold. These results showed that after light curing, additional cross-linking reactions of resin components could still be in progress, thereby leading to increases in flexural strength and modulus[36].

At the immediate condition, with an exception of Point 4 pair, all materials showed lower flexural strengths than the control. After one month or longer, FCRs exhibited flexural strengths which were 80−140% of the control. This result was remarkable, since it was reported that the strengths of FCRs were about 60−90% of those of CCRs[1].

Weibull analysis showed that although the Weibull moduli of FCRs could be similar to those of CCRs, their characteristic strengths were signifi-

cantly different. High values of Weibull modulus (5.01−14.30) showed that the findings were quite reliable when compared with the results from other specimen configurations[18]. After one year, the Weibull moduli of the materials slightly fluctuated. However, it was not a wide variance for each material.

It was reported that Weibull analysis could be used to predict the probability of failure under any level of flexural stress application[14]. In the opposite sense, the stress level at which 10−20% of the materials will be fractured — which is typically deemed as clinical failure benchmark[18] — can be calculated using Weibull cumulative Gaussian plots, without due consideration of the mean values. For example, after one year in water storage, the material in this study that needed the highest force to break 10% of the specimens was Unifil Flow, which required a stress level of 134.60 MPa (Fig. 1A). This value was higher than its counterpart, Unifil F, which needed only a stress value of 50.42 MPa (Fig. 1B). It was also higher than the control, Herculite XRV, which needed a stress value of 86.74 MPa (Fig. 1C). The prediction of fracture under any level of flexural stress application using the Weibull analysis confirmed the Scheffé's test result — that in general, the flexural strengths of FCRs were higher than those of CCRs after one-year water storage.

It was mentioned that with light-cured restoratives, material strength and elastic modulus were well correlated with filler content, filler size, and distribution of filler particles[3]. In this study, the presence of different filler contents, filler particle sizes, and stiffness made it difficult to compare the relation of filler contents with mechanical properties. Moreover, the shape, composition, interparticle spacing, and surface treatment of the filler might play a role in influencing the mechanical properties of composite resins[12,13]. Besides, it must be pointed out that hoop stresses exist around filler particles as a result of matrix shrinkage during polymerization. These hoop stresses increase frictional force between fillers and resin matrix, thereby decreasing filler pullout tendency during flexural testing[28,29,36]. Although it was reported that composites containing barium glass showed a greater tendency to degrade in water than would the composites that contained quartz[32], this tendency was not noticeable in this study.

Previously, it was mentioned that flowability be mainly achieved by lowering the filler loading[1]. According to the information provided by the manufacturers, FCRs had a lower filler content than CCRs, except for Filtek Flow which had a higher filler content than Filtek A110. Then, except for Metafil C-Metafil Flo pair, the flexural moduli of FCRs were significantly lower than the CCRs'. The unusual pattern showed by the Metafil pair was still unclear. Notwithstanding this exception, it could be said that the low stiffness of FCRs compensated for the polymerization contraction of the higher-modulus restorative composite materials[37].

After storage, regardless of their flowability, the flexural moduli of the materials increased, which meant that the stiffness of both FCRs and CCRs increased. Since FCRs had a lower filler content, they also shrank more — which meant that there was a high potential for interfacial stresses to be exerted on the bonding agents during composite curing[38]. However, the application of FCR still has a prominent advantage. Traditional hybrid composites present greater marginal voids and microleakage than FCRs[39]. Thus, the choice and use of an appropriate application technique, such as incremental filling technique, should be employed to provide better internal adaptation to the tooth structure[8,40] and to reduce polymerization shrinkage stresses.

Another parameter that describes the flexural property is modulus of resilience. It refers to the amount of energy stored up in a body when one unit volume of material is stressed to its proportional limit[11]. If little energy is needed to break a material, then cracks are more likely to be formed more readily, thus resulting in increased wear rate. In this study, except for Point 4 pair, FCRs exhibited relatively higher moduli of resilience than the CCRs. Hence, the FCRs would have higher wear resistance than the CCRs[21]. In comparison with the immediate condition, the materials showed no significant differences or a slight decrease in their modulus of resilience values. This meant that after one year of static water storage, the durability of FCRs was still better than the CCRs', although less energy was needed to break both types of materials. This was an important finding and a big boost for FCRs, since previous study revealed no significant differences in wear rate during tooth abrasion between FCR and condensable composite[1].

One factor that influences the modulus of resilience and which also partially compensates for polymerization shrinkage is water sorption[24]. To study the trend of water sorption in a long-term period, it is useful to plot a fitting curve (Fig. 2). Although there were some potential equations that could represent the water sorption cumulative data, only two equations could fit and create an excellent sigmoidal fitting curve. Further, although the Chapman's equation was slightly better than the Hill's, the water sorption equation is an interesting subject that warrants further study, especially when the specimen geometry was not a disk as stated in the ISO.

The resin matrix of the composites was believed

to have absorbed water — usually in a small percentage, and which then changed the magnitude of some physical properties[41]. Differences in water sorption values also stemmed from differences in the following aspects: chemical composition of the matrix monomers, diffusion coefficient and boundary conditions at the surface of material, pore size and pore volume, and the rate of polymerization[13,42-44].

Metafil Flo exhibited the lowest water sorption rate among the FCRs, but that of Metafil C was in the mid range of CCRs. This difference could be attributed to the nanofiller content of Metafil Flo *versus* the microfiller content of Metafil C. As for Unifil F, tit contained fluoride and exhibited the highest water sorption rate. This was because the material must first absorb water in order to initiate the release of fluoride[44]. However, water softened composites by penetrating the matrix and caused unreacted monomer and unbound components to leach[17,23]. With a higher water sorption rate, a lower amount of energy was needed to break the material[41], as shown by the modulus of resilience value. However, this statement requires further consideration, since Unifil F which had the highest water sorption rate did not show the lowest flexural strength among the materials in this study.

As mentioned before, flexural strength and modulus of resilience are well correlated with quantitative clinical wear data[21]. Specifically, flexural strength and modulus of resilience seem to reflect *in vivo* wear performances. The fracture toughness of FCRs is higher than that of packable composite resins[46], and the fracture toughness of packable resins is higher than or not significantly different from that of CCRs[47]. In view of these results, it could be said that FCRs are more resistant to crack propagation than CCRs.

In conclusion, most of the investigated FCRs showed higher flexural strength, higher durability, and lower water sorption than their CCR counterparts in a one-year observation. It was demonstrated that within each FCR-CCR pair, both types of materials exhibited very different characteristics and mechanical properties, as they differed in filler and base monomer contents.

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Flexural Performance of Flowable versus Conventional Light-cured Composite Resins in a Long-term in vitro Study

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Flexural Performance of Flowable *versus* Conventional Light-cured Composite Resins in a Long-term *in vitro* Study

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The purpose of this study was to evaluate the flexural strength, flexural modulus, modulus of resilience, and water sorption of four flowable light-cured composite resins (FCRs). Results were then compared with four conventional composite resins (CCRs) and a minifilled hybrid light-cured composite resin, which served as a control. Twenty specimens were flexural tested immediately after curing, while others were stored in water at 37℃ for 1 month, 3 months, 6 months, or 1 year before flexural testing. The 1-year specimens were weight-measured at designated time intervals to examine water sorption. All FCRs, except Point 4 Flowable, showed higher flexural strength values than their CCR counterparts ($p<0.05$, Scheffé's test). After one-year water storage, the flexural strengths and flexural moduli of FCRs increased 1.5-fold or more when compared with the immediate condition. In most cases, the moduli of resilience of FCRs were higher than those of CCRs. In conclusion, it was found that FCR and CCR with the same brand name had very different characteristics and mechanical properties.

Keywords: Flowable composite, Mechanical property, Long-term durability

INTRODUCTION

Resin composite formulations with easy-to-flow characteristic have been introduced as the so-called 'flowable' composite resins (FCRs). Bayne *et al.* reported that the mechanical properties of several commercial FCRs were about 60% to 90% of those of conventional composite resins (CCRs)[1]. Therefore, FCR is usually used as a liner in areas of difficult access or flow such as irregular internal surfaces and proximal boxes of Class II preparations. This is because FCR easily flows into, adapts to, and fills the tooth surface, resulting in less leakage and hence less internal restorations with voids and post-operative sensitivity[1-7]. However, in restorations prepared by sandwich technique, gaps still exist between the FCR liner and the hybrid composite filling material[8].

Despite limited scientific information, FCRs are used in a wide range of applications[9,10]. Nonetheless, dentists are uncertain and divided if FCR can be used as a complete restoration. This is because caution is already exercised on the amount of FCR to be used in posterior restorations to avoid problems arising from the poor mechanical properties of FCRs[8]. Meanwhile, owing to strong tides of continuous research development and high demand for FCRs, some manufacturers have ridden on the success of their CCR products and launched the FCR products. Despite the deluge of FCR products that have entered the market, no studies have been undertaken to compare FCRs with their CCR counterparts of the same brand name.

In the oral environment, FCRs must withstand masticatory and parafunctional stresses. They must maintain their integrity while transferring stresses to the tooth structure. It is also noteworthy that these stresses vary markedly in different clinical situations. Consequently, threshold values for mechanical properties — which were suggested to ensure long-term success of dental restorations — may vary considerably from one case to another. In cases where greater stresses are anticipated, then stronger FCRs are required. Therefore, with a view to unraveling the real intrinsic characteristics of FCRs, it is preferable to measure the strength of the FCR *per se*, without being placed in the tooth cavity and thus inviting the complicating presence of adhesive materials.

To determine if a composite resin filling material can resist masticatory force, flexural strength is one of the mechanical properties that must be assessed[11-13]. As a collective measurement of tensile, compressive and shear stresses simultaneously, flexural strength measurement is used to evaluate the fracture resistance and elasticity of a material[12,13]. Then, for the analysis of flexural strength data, Weibull distribution analysis has been recommended. This is because it is capable of allowing for skewed data and predicting values within and outside the data set[4,14-19]. By measuring flexural strength, the

flexural modulus and modulus of resilience are measured too. Flexural modulus describes the stiffness of a material, since marginal breakdown and loss of marginal seal are most likely to occur in products with a lower modulus of elasticity[1,17,20]. Then, the energy needed to break a material is expressed by the modulus of resilience[11,22,23].

The flexural properties of composite resins are also time-dependent. As the polymerization of composites progresses even after being light-cured[17,23], an incomplete polymerization may predispose resin restoratives to degradation. Unreacted molecules can form walls of pores within the bulk material, which can be filled with water and cause water sorption[11]. However, this characteristic can be advantageous to FCR as a partial compensation for polymerization shrinkage[24]. For this reason, the long-term flexural characteristics of FCR must be studied because of important clinical implications. Besides, physical properties — such as water sorption that plays an important role in the long-term success of restorative materials — must be considered too[22,25].

Therefore, this investigation was carried out to evaluate the long-term flexural strength, flexural modulus, modulus of resilience, and water sorption of FCR. The hypothesis tested was that the properties of FCR would be significantly different from those of CCR.

MATERIALS AND METHODS

Materials used

Four FCRs (Metafil Flo, Filtek Flow, Unifil Flow, and Point 4 Flowable) and four CCRs (Metafil C, Filtek

Table 1 Materials investigated in this study. Information is as provided by the manufacturers

Material (Manufacturer)	Batch no.	Filler content	Monomer	Curing time (seconds)
Metafil Flo* (Sun Medical, Moriyama, Japan)	VV10, VV12, EK1, EK2	Barium silica glass, colloidal silica & TMPT Filler content: 44 vol% (65 wt%) Filler particle size: 0.01−10 μm	UDMA	40
Metafil C** (Sun Medical, Moriyama, Japan)	TE1	TMPT & colloidal silica Filler content: 54 vol% (66 wt%) Filler particle size: 0.01−10 μm	UDMA	40
Filtek™ Flow* (3M, St. Paul, MN, USA)	OBK, 20010104	Silica & silica zirconia Filler content: 47 vol% (68 wt%) Filler particle size: 1.50 μm	Bis-GMA, TEGDMA	20
Filtek A 110** (3M, St. Paul, MN, USA)	1AP	Inorganic silica Filler content: 40 vol% (56 wt%) Filler particle size: 0.04 μm	Bis-GMA	40
Point 4 Flowable* (Kerr, Orange, CA, USA)	206B43	Barium silica glass Filler content: 48 vol% (70 wt%)	TEGDMA, EBPADM	40
Point 4** (Kerr, Orange, CA, USA)	205553	Barium aluminoborosilicate glass Filler content: 57 vol% (76 wt%) Filler particle size: 0.4 μm	Bis-GMA, TEGDMA & EBPADM	40
Unifil Flow* (GC Corp. Tokyo, Japan)	0107201	Fluoro-alumino-silicate, silica Filler content: 67 wt% (vol%: Not Available) Filler particle size: 0.7 μm	UDMA (26%) Dimethacrylate (7%)	40
Unifil F** (GC Corp. Tokyo, Japan)	161181, 0204031	Fluoro-alumino-silicate, silica Filler content: 77 wt% (vol%: Not Available) Filler particle size: 0.8−0.9 μm	UDMA (16%) Dimethacrylate (7%)	20
Herculite XRV** Kerr, Orange, CA, USA	112330	Barium silicate Filler content: 59 vol% (78.8 wt%) Filler particle size: 0.6 μm	Bis-GMA, TEGDMA	40

* : Flowable type (FCR), ** : Conventional type (CCR) ; UDMA=Urethane dimethacrylate; Bis-GMA=Bisphenyl glycidyl-methacrylate ; TEGDMA=Triethylene glycol-dimethacrylate; EBPADM=Ethoxylated bis-phenol A dimethacrylate

A110, Unifil F, and Point 4) of Shade A3, paired from the same manufacturers, were used in this study. A minifilled hybrid light-cured composite resin (Herculite XRV) was used in this study as a control material. Details of the materials, as provided by the manufacturers, are listed in Table 1.

A visible light curing unit (New Light VL-II, GC Corp., Tokyo, Japan) with an irradiated diameter of 10 mm was used for activating the specimens. Close contact between exit window of the lamp and celluloid strip was ensured. Light intensity was checked and maintained at 450 mW/cm^2 using a radiometer (Demetron/Kerr, Danbury, CT, USA).

Flexural strength measurement

Internal dimensions of the Teflon split molds used in this study were 25×2×2 mm. A total of 100 specimens were prepared for each material. Each mold was filled with the material, covered with a celluloid strip, and then the glass plate clamped. After 15 seconds, the glass plate was removed and the specimen was light-cured in three overlapping sections with an irradiation time of 20, 30, or 40 seconds according to manufacturer's recommendations. After the specimens were removed from the molds, excess material was removed with a silicon carbide bur. Following which, the specimens were polished with #600 sandpaper to acquire flat surfaces.

Twenty specimens were flexural tested immediately, while others were immersed in 37℃ distilled water in an incubator for the following storage periods before flexural testing: 1 month, 3 months, 6 months, or 1 year. For each material and the designated storage period, 20 specimens were prepared. Prior to testing, the specimens were measured using a digital micrometer (No. 293-421-20, Mitsutoyo, Kawasaki, Japan).

Using a three-point bending method with a 20-mm span and a crosshead speed of 0.5 mm/min, flexural strength was measured by mounting the apparatus on a universal testing machine (Autograph DCS-2000, Shimadzu, Kyoto, Japan) as outlined in ISO 9917-2 (1996)[22,26]. Then, a maximum external force of 10 kgf (98 N) was applied to the middle of the test beam.

From the flexural strength result, flexural modulus in MPa was calculated and subsequently converted to GPa[9,18,23]. Then, using the values of flexural strength and flexural modulus, the modulus of resilience was computed[11,21].

All procedures, except for mechanical testing, were performed in an air-conditioned room at 23±0.5℃ and 50±2% relative humidity.

Change in weight after 24 hour/water sorption

To assess the degree of water sorption, specimens for 1-year flexural strength measurement were weighed — prior to immersion in distilled water — with an electric balance (AJ 100, Mettler, Greifensee, Switzerland). The specimens were weighed again after being stored in 37℃ distilled water for 24 hours and at several time intervals up to one year. Prior to weight measurement, the specimens were dried for one minute on Kim Wiper. Changes in specimen's weight between the immediate condition (*i.e.*, immediately after light curing) and after water storage were expressed as a percentage[22].

Statistical analysis

Flexural strength and flexural modulus results were analyzed statistically using three-way ANOVA and Scheffé's test[27] at a significance level of 0.05. Flexural strength test results were also analyzed statistically using Weibull statistics with the following equation:

$$Pf = 1 - \exp[-(\sigma / \sigma_o)m]$$

where Pf is the probability of failure, σ is the strength at a given Pf, σ_o is the characteristic strength, and m is the Weibull modulus, a constant factor related to the dispersion of failure data[14,15,17].

Results of water sorption test were also analyzed statistically using three-way ANOVA and Scheffé's test[27]. Subsequently, using Sigma Plot 8.0 program (SPSS, Chicago, IL, USA), the data were fitted to a non-linear curve by plotting water sorption (percentage) as a function of time (days).

RESULTS

Flexural strength

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the flexural strength of composite resins significantly ($p<0.001$). Table 2 lists the flexural strength results of all the materials tested in this study. Scheffé's test between the pairs and among the materials within the same water storage period showed significant differences ($p<0.05$). In the immediate condition, all FCRs would be completely fractured by a flexural stress of 90 MPa, with the mean value of Point 4 Flowable > Metafil Flo > Unifil Flow > Filtek Flow. All FCRs, except Point 4 Flowable, showed higher flexural strength values than their CCR counterparts, and that this situation continued up to one year. Predicting from Weibull cumulative Gaussian plots, such as those of Unifil pair and Herculite XRV (Fig. 1), it could be seen that for all 1-year FCR specimens, a minimum of 110 MPa flexural stress must be applied before fracture would occur. On the contrary, 110 MPa was the maximum strength required for fracturing the CCRs.

Weibull analysis (Table 3) showed that Weibull moduli varied from 5.01 to 14.30. Except for Point 4 Flowable, the characteristic strengths of CCRs were lower than those of FCRs. Further, statistical analysis showed high coefficients of correlation (r), varying between 0.96 and 0.98.

Flexural modulus

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the flexural modulus of composite resins significantly ($p<0.001$). In the immediate condition, as shown in Table 4, the flexural moduli of FCRs were in the range of 1.38−3.16 GPa. Those of CCRs were between 2.03 and 3.71 GPa, and that of control was 5.62 GPa. After one year, the flexural moduli increased. The flexural moduli of FCRs increased to a range of 5.51−7.47 GPa, while those of their CCR counterparts were between 4.53 and 8.89 GPa, and that of Herculite XRV was 9.50 GPa. Scheffé's test showed significant differences in flexural modulus within each CCR-FCR pair ($p<0.05$). In general, the flexural moduli of Unifil F, Filtek A110, and Herculite XRV were higher than those of Unifil Flow and Filtek Flow. However, in most conditions, the flexural modulus of Metafil C was lower than that of Metafil Flo.

Modulus of resilience

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the modulus of resilience of composite resins significantly ($p<0.001$). As shown in Table 5, Scheffé's test for the modulus of resilience showed that FCRs had higher modulus of resilience values than CCRs. Further, throughout the one-year period, all FCRs showed almost no significant differences among their values. The moduli of resilience of FCRs varied between 0.62 MJ/m^3 and 1.93 MJ/m^3, while those of CCRs varied between 0.20 MJ/m^3 and 1.21 MJ/m^3.

Change in weight after 24 hours/water sorption

ANOVA showed that differences in the type and maker of composite resin and a long immersion period in distilled water would affect the water sorption of composite resins significantly ($p<0.001$). The results of water sorption are listed in Table 6. After one-year storage, all FCRs had water sorption values between 0.39% and 1.18%, while the CCRs showed water sorption values between 0.98% and 2.96%. All FCRs, except Point 4 Flowable, showed significantly lower water sorption values than their CCRs ($p<0.05$), and that Unifil F showed the highest water sorption value among the CCRs (Fig. 2). The water

Table 2 Mean values of flexural strength of composite resins (Mean(SD) in MPa)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	75.69 (7.35) $^{C,\omega,\zeta}$	127.27 (14.35) $^{B,\rho,\sigma}$	119.91 (12.77) $^{B,\kappa,\lambda}$	77.73 (8.65) $^{C,\iota}$	142.37 (12.47) $^{A,\alpha}$
Metafil C**	50.10 (60.2) $^{E,\#}$	76.91 (4.22) $^{C,\tau}$	67.56 (8.19) $^{C,D,o,\pi}$	60.81 (8.11) $^{D,E,\phi}$	75.82 (10.46) $^{C,\zeta,\delta}$
Filtek ™ Flow*	62.42 (10.29) $^{G,\Psi,\zeta}$	111.25 (13.44) $^{F,\sigma}$	106.08 (11.94) $^{F,\mu}$	113.33 (13.23) $^{F,\gamma,\eta}$	112.59 (9.90) $^{F,\beta}$
Filtek A 110**	55.44 (7.70) $^{G,\zeta,\#}$	52.52 (5.82) $^{G,\upsilon}$	51.09 (7.63) $^{G,\pi,\theta}$	61.01 (6.17) $^{G,\phi}$	48.71 (5.91) ^{G,s}
Point 4 Flowable*	86.88 (11.46) $^{I,\varpi,\omega}$	124.63 (17.90) $^{H,\rho,\sigma}$	88.69 (14.02) $^{I,\mu,\nu}$	90.63 (12.19) $^{I,\eta,\iota}$	88.83 (11.77) $^{I,\delta}$
Point 4**	88.02 (13.22) $^{I,\varpi}$	129.12 (16.68) $^{H,\rho}$	129.22 (20.29) $^{H,\kappa}$	117.59 (16.40) $^{H,\gamma}$	118.52 (20.16) $^{H,\beta}$
Unifil Flow*	71.37 (5.93) $^{L,M,\xi,\Psi}$	126.70 (13.59) $^{K,\rho,\sigma}$	132.30 (20.46) $^{K,\kappa}$	136.82 (16.97) $^{J,K,\phi}$	153.08 (11.90) $^{J,\alpha}$
Unifil F**	67.88 (10.37) $^{M,\xi,\Psi}$	87.55 (17.31) $^{L,\tau}$	78.37 (12.80) $^{L,M,\nu,o}$	60.86 (12.82) $^{M,\phi}$	68.02 (11.72) $^{M,\delta}$
Herculite XRV**	90.20 (11.28) $^{R,\varpi}$	119.20 (15.46) $^{P,\rho,\sigma}$	108.38 (15.96) $^{P,Q,\lambda}$	97.22 (16.77) $^{Q,R,\eta}$	115.29 (18.99) $^{P,\beta}$

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin

Identidal capital letters indicate no significant difference ($p>0.05$, Scheffé's test) analyzed between pairs and among the values of control

Identical Greek letters and # indicate no significant difference ($p>0.05$, Scheffé's test) analyzed among the values of the same period

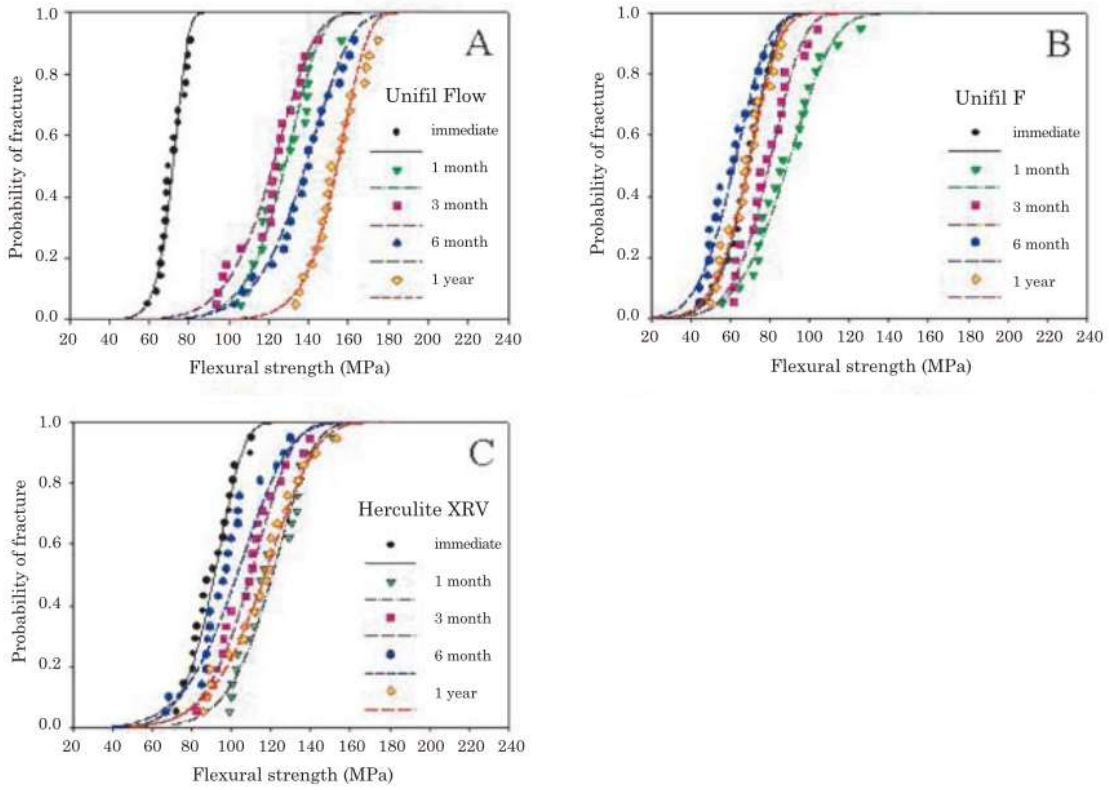

Fig. 1 Weibull cumulative Gaussian plots for Unifil pair and Herculite XRV.

Table 3 Characteristic strengths (MPa) and Weibull moduli of composite resins

Material	Immediate		1 month		3 month		6 month		1 year	
	CS	WM	CS	WM	CS	WM	CS	WM	CS	WM
Metafil Flo*	79.08	10.35	133.69	9.47	125.67	9.98	82.51	8.68	148.16	12.04
Metafil C**	52.80	8.82	78.59	10.98	71.30	8.62	64.69	7.34	80.51	7.57
Filtek ™ Flow*	66.89	6.37	117.73	8.18	111.44	9.45	119.26	9.10	117.08	12.09
Filtek A 110**	58.90	7.47	55.14	9.58	54.40	7.16	63.88	10.25	51.45	8.41
Point 4 Flowable*	92.02	7.93	132.41	7.45	94.82	6.64	96.21	7.62	94.08	7.97
Point 4**	93.87	6.96	136.47	8.29	138.04	6.71	124.81	7.58	127.21	6.20
Unifil Flow*	74.13	12.70	132.94	9.80	141.19	6.85	144.52	8.38	158.58	13.73
Unifil F**	72.49	6.75	94.74	5.36	83.95	6.44	66.18	5.01	73.10	6.06
Herculite XRV**	95.22	8.50	126.21	8.06	115.30	7.26	109.69	5.54	123.71	6.34

n=20 ; CS=characteristic strength; WM=Weibull modulus
*=flowable light-cured composite resin; **=conventional light-cured composite resin

Table 4 Mean values of flexural modulus of composite resins (Mean(SD) in GPa)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	1.95 (0.22) E, δ, ε	5.04 (0.42) B, κ	5.06 (0.43) B, θ, ρ	3.31 (0.23) D, ξ	7.47 (0.67) A, ζ
Metafil C**	2.03 (0.17) E, δ	4.36 (0.31) C, κ, λ	4.02 (0.28) C, σ	3.42 (0.20) D, ξ	4.53 (0.27) B, C, ¥
Filtek ™ Flow*	1.38 (0.09) M, φ	4.32 (0.26) H, λ	4.55 (0.41) I, ρ, σ	4.87 (0.74) H, ω	5.51 (0.43) I, J, $
Filtek A 110**	3.55 (0.28) L, β, δ	7.12 (0.30) K, ι	6.17 (0.57) K, π	6.99 (0.26) K, ϖ	6.05 (0.11) I, #, $
Point 4 Flowable*	3.16 (0.38) N, δ	6.39 (0.51) T, φ	5.65 (0.35) U, π, θ	6.68 (0.41) S, T, ϖ	6.45 (0.48) S, T, #
Point 4**	3.71 (0.40) N, β	7.68 (0.65) Q, R, ι	7.10 (0.61) R, S, ο	8.15 (0.67) Q, υ	8.87 (0.59) P, Ψ
Unifil Flow*	1.47 (0.27) Z, S, φ	4.98 (0.57) Y, κ	5.34 (0.85) X, Y, θ	5.52 (0.45) X, Y, ω	6.24 (0.62) X, #
Unifil F**	2.24 (0.27) Z, δ	8.49 (0.92) W, η	8.34 (0.99) W, ν	9.84 (1.19) V, τ	8.89 (0.82) W, Ψ
Herculite XRV**	5.62 (0.50) F, α	9.60 (0.61) G, γ	9.54 (0.54) G, μ	9.18 (0.93) G, τ	9.50 (0.51) G, Ψ

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant difference (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters and symbols ($,¥, #) indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values of the same period

Table 5 Mean values of the modulus of resilience of composite resins (Mean(SD) in MJ/m^3)

Material	Immediate	1 month	3 month	6 month	1 year
Metafil Flo*	1.50 (0.31) A, α, β	1.65 (0.38) A, φ	1.45 (0.37) A, κ, λ	0.93 (0.18) B, ρ	1.35 (0.28) A, ξ
Metafil C**	0.65 (0.12) B, C, ε	0.68 (0.11) B, C, ι	0.58 (0.15) C, μ, ν	0.55 (0.14) C, τ	0.69 (0.14) B, C, Ψ
Filtek ™ Flow*	1.50 (0.50) E, F, α, β	1.52 (0.19) E, φ, γ	1.41 (0.28) E, F, λ	1.35 (0.06) E, F, θ	1.20 (0.26) F, ξ
Filtek A 110**	0.45 (0.10) G, ε	0.20 (0.03) G, φ	0.24 (0.06) G, ο	0.28 (0.04) G, υ, π	0.20 (0.03) G, ζ
Point 4 Flowable*	1.24 (0.43) H, I, β, π	1.25 (0.38) H, γ, η	0.80 (0.17) J, K, μ	0.63 (0.17) K, σ, τ	0.62 (0.18) K, Ψ
Point 4**	1.05 (0.33) H, I, J, K, π, δ	1.13 (0.37) H, I, J, η	1.21 (0.37) H, I, j, λ	0.86 (0.22) I, J, K, ρ, σ	0.81 (0.29) J, K, Ψ
Unifil Flow*	1.82 (0.37) L, a	1.66 (0.47) L, φ	1.74 (0.25) L, κ	1.71 (0.36) L, π	1.93 (0.39) L, ω
Unifil F**	1.07 (0.36) M, β, π, δ	0.47 (0.18) N, ι, φ	0.38 (0.15) N, ν, ο	0.22 (0.10) N, ϖ	0.27 (0.08) N, ζ
Herculite XRV**	0.74 (0.20) P, Q, δ, ε	0.75 (0.20) P, ι	0.63 (0.20) P, Q, μ, ν	0.54 (0.21) Q, τ, υ	0.72 (0.24) P, Q, Ψ

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant dirrence (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values same period

Table 6 Mean values of water sorption of composite resins (Mean(SD) in %)

Material	1 month	3 month	6 month	1 year
Metafil Flo*	0.37 (0.04) A, γ	0.38 (0.03) A, ν	0.39 (0.03) A, ϖ	0.39 (0.03) A, #
Metafil C**	1.48 (0.10) B, δ	1.48 (0.10) B, φ	1.50 (0.09) B, θ	1.50 (0.09) B, Ψ
Filtek ™ Flow*	0.96 (0.40) G, ε	0.97 (0.05) G, λ	0.99 (0.05) G, σ	0.99 (0.04) G, ¥
Filtek A 110**	1.58 (0.11) L, β	1.69 (0.12) L, ι	1.84 (0.10) K, π	1.98 (0.14) K, ξ
Point 4 Flowable*	0.72 (0.04) Q, R, φ	0.80 (0.08) P, Q, μ	0.86 (0.07) N, P, τ	0.93 (0.08) M, N, ¥
Point 4**	0.66 (0.55) R, φ	0.77 (0.04) Q, μ	0.79 (0.06) P, Q, τ, ν	0.98 (0.05) M, ¥
Unifil Flow*	1.14 (0.04) V, δ	1.17 (0.04) V, κ	1.18 (0.03) V, ρ	1.18 (0.03) V, ζ
Unifil F**	2.81 (0.09) Y, α	2.95 (0.13) Z, η	2.95 (0.13) Z, ο	2.96 (0.11) Z, ω
Herculite XRV**	0.64 (0.05) C, φ	0.70 (0.06) C, D, μ	0.74 (0.08) D, F, υ	0.80 (0.07) F, $

n=20 ; *=flowable light-cured composite resin ; **=conventional light-cured composite resin
Identidal capital letters indicate no significant difference (p>0.05, Scheffé's test) analyzed between pairs and among the values of control
Identical Greek letters and symbols ($, ¥, #) indicate no significant difference (p>0.05, Scheffé's test) analyzed among the values of the same period

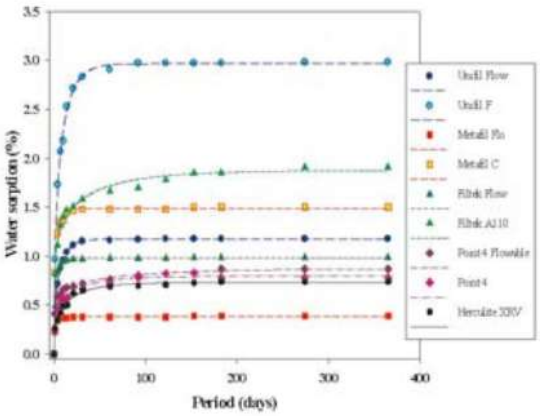

Fig. 2 Water sorption trends of FCRs and CCRs during one year of water storage.

sorption values correlated with the period of water immersion time and could be fitted to a sigmoidal curve of Chapman's three-parameter equation [$y = a\times(1-exp(-bx))^c$] or Hill's three-parameter equation [$y = ax^b/(c^b + x^b)$], with coefficient of correlation r≥0.997.

DISCUSSION

Measurement of long-term flexural strength is very important for a composite resin, since it describes the durability of the material. The degradation of composite resins in the oral environment is considered to be attributed to the resin matrix, filler particles, and hydrolytic instability of silane coupling agent at the polymer-silica interface[28-35]. After one-year water storage in the present study, all composites showed higher flexural strengths in comparison with their immediate values. FCRs increased their strengths by up to 2.14-fold, whereas their CCR counterparts increased by only up to 1.54-fold. These results showed that after light curing, additional cross-linking reactions of resin components could still be in progress, thereby leading to increases in flexural strength and modulus[36].

At the immediate condition, with an exception of Point 4 pair, all materials showed lower flexural strengths than the control. After one month or longer, FCRs exhibited flexural strengths which were 80−140% of the control. This result was remarkable, since it was reported that the strengths of FCRs were about 60−90% of those of CCRs[1].

Weibull analysis showed that although the Weibull moduli of FCRs could be similar to those of CCRs, their characteristic strengths were signifi-

cantly different. High values of Weibull modulus (5.01−14.30) showed that the findings were quite reliable when compared with the results from other specimen configurations[18]. After one year, the Weibull moduli of the materials slightly fluctuated. However, it was not a wide variance for each material.

It was reported that Weibull analysis could be used to predict the probability of failure under any level of flexural stress application[14]. In the opposite sense, the stress level at which 10−20% of the materials will be fractured — which is typically deemed as clinical failure benchmark[18] — can be calculated using Weibull cumulative Gaussian plots, without due consideration of the mean values. For example, after one year in water storage, the material in this study that needed the highest force to break 10% of the specimens was Unifil Flow, which required a stress level of 134.60 MPa (Fig. 1A). This value was higher than its counterpart, Unifil F, which needed only a stress value of 50.42 MPa (Fig. 1B). It was also higher than the control, Herculite XRV, which needed a stress value of 86.74 MPa (Fig. 1C). The prediction of fracture under any level of flexural stress application using the Weibull analysis confirmed the Scheffé's test result — that in general, the flexural strengths of FCRs were higher than those of CCRs after one-year water storage.

It was mentioned that with light-cured restoratives, material strength and elastic modulus were well correlated with filler content, filler size, and distribution of filler particles[3]. In this study, the presence of different filler contents, filler particle sizes, and stiffness made it difficult to compare the relation of filler contents with mechanical properties. Moreover, the shape, composition, interparticle spacing, and surface treatment of the filler might play a role in influencing the mechanical properties of composite resins[12,13]. Besides, it must be pointed out that hoop stresses exist around filler particles as a result of matrix shrinkage during polymerization. These hoop stresses increase frictional force between fillers and resin matrix, thereby decreasing filler pullout tendency during flexural testing[28,29,36]. Although it was reported that composites containing barium glass showed a greater tendency to degrade in water than would the composites that contained quartz[32], this tendency was not noticeable in this study.

Previously, it was mentioned that flowability be mainly achieved by lowering the filler loading[1]. According to the information provided by the manufacturers, FCRs had a lower filler content than CCRs, except for Filtek Flow which had a higher filler content than Filtek A110. Then, except for Metafil C-Metafil Flo pair, the flexural moduli of FCRs were significantly lower than the CCRs'. The unusual pattern showed by the Metafil pair was still unclear. Notwithstanding this exception, it could be said that the low stiffness of FCRs compensated for the polymerization contraction of the higher-modulus restorative composite materials[37].

After storage, regardless of their flowability, the flexural moduli of the materials increased, which meant that the stiffness of both FCRs and CCRs increased. Since FCRs had a lower filler content, they also shrank more — which meant that there was a high potential for interfacial stresses to be exerted on the bonding agents during composite curing[38]. However, the application of FCR still has a prominent advantage. Traditional hybrid composites present greater marginal voids and microleakage than FCRs[39]. Thus, the choice and use of an appropriate application technique, such as incremental filling technique, should be employed to provide better internal adaptation to the tooth structure[8,40] and to reduce polymerization shrinkage stresses.

Another parameter that describes the flexural property is modulus of resilience. It refers to the amount of energy stored up in a body when one unit volume of material is stressed to its proportional limit[11]. If little energy is needed to break a material, then cracks are more likely to be formed more readily, thus resulting in increased wear rate. In this study, except for Point 4 pair, FCRs exhibited relatively higher moduli of resilience than the CCRs. Hence, the FCRs would have higher wear resistance than the CCRs[21]. In comparison with the immediate condition, the materials showed no significant differences or a slight decrease in their modulus of resilience values. This meant that after one year of static water storage, the durability of FCRs was still better than the CCRs', although less energy was needed to break both types of materials. This was an important finding and a big boost for FCRs, since previous study revealed no significant differences in wear rate during tooth abrasion between FCR and condensable composite[1].

One factor that influences the modulus of resilience and which also partially compensates for polymerization shrinkage is water sorption[24]. To study the trend of water sorption in a long-term period, it is useful to plot a fitting curve (Fig. 2). Although there were some potential equations that could represent the water sorption cumulative data, only two equations could fit and create an excellent sigmoidal fitting curve. Further, although the Chapman's equation was slightly better than the Hill's, the water sorption equation is an interesting subject that warrants further study, especially when the specimen geometry was not a disk as stated in the ISO.

The resin matrix of the composites was believed

to have absorbed water — usually in a small percentage, and which then changed the magnitude of some physical properties[41]. Differences in water sorption values also stemmed from differences in the following aspects: chemical composition of the matrix monomers, diffusion coefficient and boundary conditions at the surface of material, pore size and pore volume, and the rate of polymerization[13,42-44].

Metafil Flo exhibited the lowest water sorption rate among the FCRs, but that of Metafil C was in the mid range of CCRs. This difference could be attributed to the nanofiller content of Metafil Flo *versus* the microfiller content of Metafil C. As for Unifil F, tit contained fluoride and exhibited the highest water sorption rate. This was because the material must first absorb water in order to initiate the release of fluoride[44]. However, water softened composites by penetrating the matrix and caused unreacted monomer and unbound components to leach[17,23]. With a higher water sorption rate, a lower amount of energy was needed to break the material[41], as shown by the modulus of resilience value. However, this statement requires further consideration, since Unifil F which had the highest water sorption rate did not show the lowest flexural strength among the materials in this study.

As mentioned before, flexural strength and modulus of resilience are well correlated with quantitative clinical wear data[21]. Specifically, flexural strength and modulus of resilience seem to reflect *in vivo* wear performances. The fracture toughness of FCRs is higher than that of packable composite resins[46], and the fracture toughness of packable resins is higher than or not significantly different from that of CCRs[47]. In view of these results, it could be said that FCRs are more resistant to crack propagation than CCRs.

In conclusion, most of the investigated FCRs showed higher flexural strength, higher durability, and lower water sorption than their CCR counterparts in a one-year observation. It was demonstrated that within each FCR-CCR pair, both types of materials exhibited very different characteristics and mechanical properties, as they differed in filler and base monomer contents.

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